



# Fabrication of porous NiAl intermetallic compounds with a hierarchical open-cell structure by combustion synthesis reaction and space holder method



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## ABSTRACT

We have developed a process to fabricate porous NiAl intermetallic compounds with a bi-modal porous structure by combining the combustion synthesis reaction and the space holder method. The fabricated porous NiAl showed large pores of the order of several tens of micrometers derived from the NaCl space holder and small pores of the order of several micrometers derived from the combustion synthesis reaction. The NaCl space holder acted as a heat absorber and contributed to the formation of small pores by preventing the melting of NiAl. Although the reaction was difficult to complete for large volume fractions of NaCl, it was possible to obtain single-phase NiAl by controlling the sintering conditions. We could finally obtain highly porous (porosity: > 80%) single-phase NiAl with a hierarchical open-cell structure.

## 1. Introduction

Open-cell porous materials possess unique properties such as fluid permeability and large surface area, which can be utilized for many popular fields. Semenik and Catton (2009) showed the application of porous materials on heat transport devices, and also Kodama et al. (2013) used porous metal on catalysts. For example, a loop heat pipe (LHP) is one of the heat transport devices not requiring electrical power. As reviewed by Maydanik (2005) and Launay et al. (2007), the driving force for LHP is the capillary force ( $P_{cap}$ ) in the porous material wick.  $P_{cap}$  is expressed by the following equation.

$$P_{cap} = \frac{2\sigma \cos \theta}{r_{pore}}, \quad (1)$$

where  $\sigma$  is the surface energy of the heat transport fluid,  $\theta$  is the contact angle, and  $r_{pore}$  is the pore radius of the porous wick. Eq. (1) shows that  $P_{cap}$  increases with decreasing pore size. On the other hand, Carman (1937, 1939) proved that the pressure loss ( $\Delta P/L$ ) of laminar flow through a solid-particle packed bed with a thickness of  $L$  is expressed by the following Kozeny-Carman equation.

$$\frac{\Delta P}{L} = \frac{180V_0\mu(1-\varepsilon)^2}{\Phi_s^2 D_p^2 \varepsilon^3}, \quad (2)$$

where  $V_0$  is the superficial velocity,  $\mu$  is the viscosity,  $\Phi_s$  is the

sphericity of the particles in the packed bed,  $D_p$  is the equivalent sphere diameter of the particles, and  $\varepsilon$  is the porosity. Eq. (2) shows that  $\Delta P/L$  can be reduced by decreasing  $D_p$  and increasing  $\varepsilon$ .  $D_p$  and  $r_{pore}$  have a positive correlation and  $\Delta P/L$  increases with decreasing  $r_{pore}$ . Thus, there is a trade-off between  $P_{cap}$  and  $\Delta P/L$ . However, a hierarchical cellular structure with bi-modal pore size distribution can solve this problem. Byon and Kim (2012) utilized spherical glass powder to fabricate bi-modal porous materials for application as a wick. They demonstrated that the control of the size ratio of the glass powder and its cluster helped achieve high capillary performance because the liquid penetrating the small pores wets the walls of the large pores and improves the capillary force. Thus, bi-modal porous materials have better hydromechanical properties than mono-modal porous materials.

Many researchers have attempted to prepare bi-modal porous materials. Byon and Kim (2012) sintered clusters of spherical glass particles. Zhang et al. (2013) combined the Gasar process with a dealloying method to obtain bi-modal porous Cu having good application prospects in fuel cells, sensors, and catalytic processes. Huang et al. (2009) fabricated bi-porous Ni alloys by coating Ni alloy powder on open-cell porous Ni. One promising method to fabricate bi-modal porous metallic materials is the combination of the combustion synthesis reaction and the space holder method. The space holder method involves production of a preform by which space holder particles are dispersed in a metal by powder sintering or liquid metal infiltration, followed by the removal of

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the space holder particles. This method enables direct and accurate control of the pore morphology and porosity. Zhao and Sun (2001) and Torres et al. (2012) fabricated porous Al and Ti, respectively, using NaCl space holder. Combustion synthesis is the process of obtaining ceramics, composites, intermetallic compounds, and so on by chemical reactions generating a large amount of heat, and the products have many pores according to Morsi (2001). When these methods are combined, large pores due to the space holder particles and small pores due to the combustion synthesis reaction are formed. Kobashi et al. (2013) and Jiao et al. (2016) successfully fabricated TiAl intermetallic compounds with a hierarchical open-cell structure by the combustion synthesis reaction between Al and Ti and the space holder method using NaCl. On the other hand, NiAl has a high melting point (1638°C), high Young's modulus, low density, and high oxidation resistance. These features make the open-cell porous NiAl a good candidate for a wick material. In the present study, the effects of the sintering conditions and the volume fraction of the NaCl space holder on the final products and the porous structure have been investigated. We have also suggested a process to obtain highly porous single-phase NiAl with a hierarchical open-cell structure.

## 2. Experimental procedure

Ni powders (purity: 99%, size: < 1  $\mu\text{m}$ ), Al powders (purity: 99.99%, size: < 2  $\mu\text{m}$ ), and NaCl space-holder powders (purity: 99.99%, size: 30–50  $\mu\text{m}$ ) were used. The morphology of these powders is shown in Fig. 1. In terms of the equilibrium phase diagram of the Ni–Al binary system presented by Okamoto (1993), the Ni and Al powders were blended in an atomic ratio of 1:1 to obtain single-phase NiAl intermetallic compound with a B2 crystal structure. Then, the space-holder NaCl powders were blended with the Ni–Al powder mixture using an automatic mixer so that the volume fractions of NaCl ( $V_{\text{NaCl}}$ ) were 0%, 20%, 40%, 60%, and 80%. The mixed powder was placed in a graphite mold (inner diameter: 10 mm, outer diameter: 20 mm, and height: 50 mm) and pre-compressed (25 MPa) to obtain a cylindrical precursor of a diameter of 10 mm and height of 5 mm. The precursor was treated by electric current sintering in vacuum (20 Pa) at a constant pressure (5 MPa). The schematic of the sintering apparatus is shown in Fig. 2. The temperature was measured by a thermocouple, which was inserted into the graphite mold at a depth of 2 mm. The temperature was increased at a rate of 0.5°C/s and held at 450°C, 550°C, 600°C, and 650°C. The holding times were 300 s, 1800 s, and 10,800 s. Subsequently, the samples were cooled to ambient temperature. The

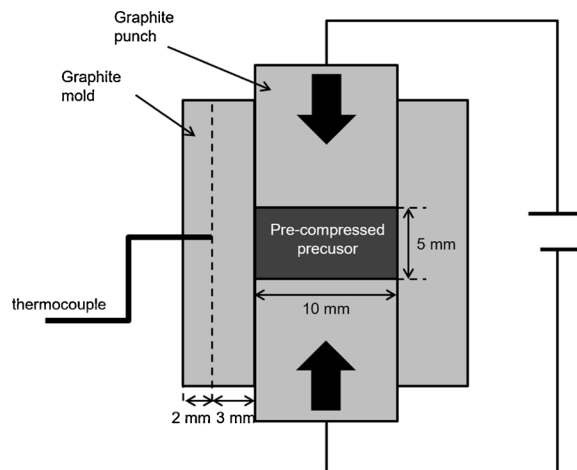


Fig. 2. Schematic of the sintering apparatus.

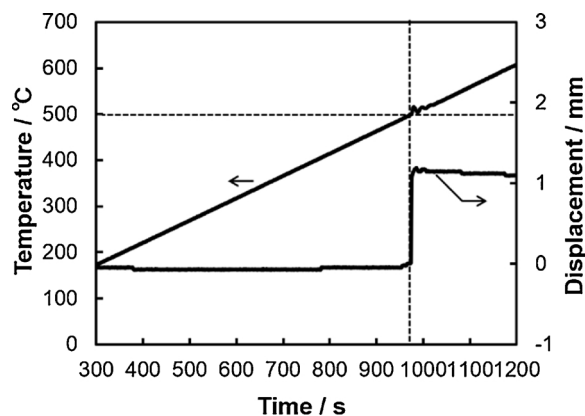


Fig. 3. Change in temperature and displacement of the graphite punch during the sintering process of Ni–Al samples.

appearance of the representative sintered sample is shown in Fig. 1. The samples were soaked in pure water for 86.4 ks to remove the NaCl space holder for the formation of large pores. For comparison, porous pure Al was fabricated by almost the same method as above. The  $V_{\text{NaCl}}$  was set at 60%. The electric current sintering was performed at 550°C for 5 min.

The masses and the sizes of the samples were measured, and the

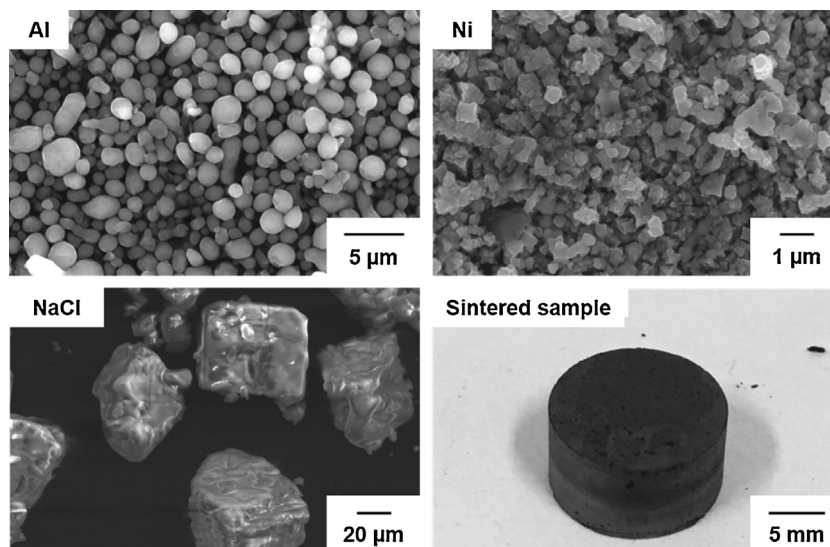


Fig. 1. SEM images of Al, Ni, and NaCl powders used in this study and the appearance of the representative sintered sample.

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